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Integrated Data Collection Analysis (IDCA) Program - Final Review September 12, 2012 at DHS

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Disclaimer

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Integrated Data Collection Analysis (IDCA) Program —Final Review September 12, 2012 at DHS

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EXCUTIVE SUMMARY

The Integrated Data Collection Analysis (IDCA) program conducted a final program review at the Department of Homeland Security on September 12, 2012. The review was focused on the results of the program over the complete performance period. The following topics were reviewed:

1. Current status of the program, including milestones, deliverables, successes and failures, summary and close out of the Proficiency Test,
2. Assessment of technical issues that arose during the Proficiency Test,
3. Recommendations for resolution of Small-Scale Safety and Thermal test issues for HMEs,
4. Potential pathway forward.

The following is an executive summary of the final review. Following in this document is a final review report that includes a more detailed summary of the technical presentations given in the review.

Current Status of Program. The Proficiency Test has essentially been completed with all 20+ materials tested by two participants; 91% of the materials tested by three participants. The IDCA has completed 23 Analysis Reports, 113 Data Reports and 10 Presentations (to outside groups).

The materials were selected for testing to understand how standard Small-Scale Safety and Thermal (SSST) testing techniques (those used for military-type materials) apply to HMEs. In addition to collecting data on 19 different materials, 12+ studies were performed where parameters were varied, through the combination of components, to further stress standard methods. This related technical issues to testing procedures. From this information, a long list of technical issues was developed.

Statistical analysis has been performed on all the materials to establish statistical relevance for both inter- and intra- laboratory comparisons. A summary of the findings is listed below.

Although one of the final products of the IDCA is to be the DHS SSST testing guide (a comprehensive collection of SSST testing data on HMEs), only a prototype document was delivered with limited data from LLNL and LANL. This document has been formatted to accept data from various sources (including the Proficiency Test) but no additional data has been added. In addition, there remain Program Analysis reports on 13 Proficiency Test materials comparing the test data among participants, that have not been completed.

Assessment of Technical Issues. HMEs have a wide variety of physical and chemical properties that most conventional explosives do not have. As a result of these properties, standard-testing methods cannot blindly be applied without consideration of modifications or revisions. The IDCA found many of the testing issues as the Proficiency Test progressed. Some of these are reported below. Note that many of these issues are reported in detail in the full reports but have not necessarily been resolved.

Testing methods. The IDCA standardized material origin, preparation and mixing. However, the testing methods for each participant were not standardized. Participants could use routine in-house testing procedures. These methods were thoroughly documented as well as any changes in methods during the performance period. Changes documented were: standardizing sandpaper grit size for impact testing (180-grit garnet), sample preparation (all powders, no pressing), revising liquid testing procedures (using and not using sandpaper for comparison), and purchasing new testing equipment. Many of these are discussed in detail below.

Comparison of Bruceton and Neyer data analysis methods. Both data analysis methods are used to determine 50% probability of reaction. The IDCA found both methods yielded about the same result on a specific material, although the Neyer method may be quicker, better for standard deviation but much more expensive.

Sandpaper affects impact testing. Sandpaper is used to hold the sample in the impact (drop hammer) test. At the beginning of the Proficiency Test, there was no specification for grit size, but widely varying results for some materials prompted standardization to a 180-grit garnet sandpaper.

ABL and BAM friction testing translation function. Although ~ 20 materials were tested for friction sensitivity by both ABL and BAM friction, no real correlation between the two sets of data was found.

ABL ESD testing compared to custom-built ESD testing. LLNL has a custom-built spark sensitivity testing system. LANL, IHD, AFRL, and SNL have various vintages of the ABL spark sensitivity testing system. Results were difficult, if not impossible, to compare between the two types of systems. LLNL purchased an ABL system, relieving this issue.

Thermal issues. Reproducibility was a real issue among the participants for differential scanning calorimetry (DSC) for most of the HMEs tested. This was not the case for RDX or PETN, where the thermal profiles are almost identical. The key issue appears to be obtaining a representative sample of mixtures. Solid-solid mixtures are often non-uniform in very small sample sizes and solid-liquid mixtures often have contact issue between the solid and the liquid during preparation and testing. Care must be taken to correctly assess the thermal sensitivity of many of the HMEs, although the lowest exothermic features were captured by all test methods so the basic thermal stability was characterized at some level.

Effect of pan type on AN decomposition. Ammonium nitrate (AN) also exhibited thermal behavior that was not reproducible among the laboratories. In addition, all the participants detected the heat flow of an obviously exothermic decomposition as an endothermic event. This was found to be due to the sample holder venting. Using a sample holder that is rated for high pressure solved the problems.

HME aging. Investigations by many DHS funded programs has shown that certain HMEs are chemically unstable and, with time, can undergo uncontrollable reactions. The effect of this aging on SSST testing has not been documented. The effect varies depending upon the HME. Some of the HMEs studied in the Proficiency Test stay the same or decrease in sensitivity upon aging, but it needs to be noted that some increase in sensitivity. Heat flow calorimetry also indicates some undergo a period of unstable exothermic behavior.

HME testing capability improvements. During the IDCA, SNL was able to procure SSST testing equipment to set up a testing facility and have it fully operational by the end of the Proficiency Test. LLNL purchased an ABL ESD testing apparatus and tested or retested about 2/3 of the materials.

Statistical evaluation. IDCA participants obtain significantly different results on many materials. The IDCA understands some of the causes—some will be hard to address without major revisions to procedures. Absolute sensitivity values are not good cross-lab comparison, while relative sensitivity rankings/order assigned by each lab are somewhat better for cross-lab comparisons.

Recommendations. The IDCA identified over 30 issues that need to be considered when applying standard SSST test methods to HMEs. These issues can adversely influence developing safe handling practices if not considered. The recommendations below are collected as potential solutions from evaluating these issues. Some recommendations are:

- Develop new sampling methods that guarantee obtaining a representative sample particularly for: very small samples of mixtures, samples that have a volatile component, and samples that have large mismatch of particle sizes,
- Carefully assess particle-size distributions of mixtures because particle size affects most measurements,
- Recognize that there is no absolute assessment of measurement data unless the safety assessment is linked to the conditions of the operation—testing conditions must reflect the operation,
- Recognize that relative sensitivity to a standard can change when testing conditions are altered, and that testing may not reflect the true sensitivity of the material for specific application,
- Develop new methods to test liquids, specifically handling the volatility issue and standards,
- Develop instrument-based detection to lessen the reliance on observation.

Pathway forward. There are many areas where additional research is needed that will help resolve some of the issues and implement the recommendations listed above:

- Understand the role of sandpaper in impact testing (relationship of sandpaper composition to particle size of HME),
- Standardize go/no-go detection (instrumentation),
- Revise DSC testing standards of materials containing liquids and include new thermal methods (volatility and reactivity),
- Revise of liquid testing methods (standards, with and without sandpaper, drop cavity),
- Standardize HME testing and handling issues (use working groups: international group for standardization—IGUS; Explosives Testers User Group—ETUG),
- Resolve statistical differences.

IDCA Explosives Safety Testing of Homemade Explosives
Integrated Data Collection Analysis Program

Integrated Data Collection Analysis (IDCA) Program —Final Review September 12, 2012 at DHS

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ABSTRACT

The Integrated Data Collection Analysis (IDCA) program conducted a final program review at the Department of Homeland Security on September 12, 2012. The review was focused on the results of the program over the complete performance period. A summary presentation delineating the accomplished tasks started the meeting, followed by technical presentations on various issues that arose during the performance period. The presentations were completed with a statistical evaluation of the testing results from all the participants in the IDCA Proficiency Test study. The meeting closed with a discussion of potential sources of funding for continuing work to resolve some of these technical issues.

This effort, funded by the Department of Homeland Security (DHS), put the issues of safe handling of these materials in perspective with standard military explosives. The study added Small-Scale Safety and Thermal (SSST) testing results for a broad suite of different HMEs to the literature, and suggested new guidelines and methods to develop safe handling practices for HMEs. Each participating testing laboratory used identical test materials and preparation methods wherever possible. Note, however, the test procedures differ among the laboratories. The results were compared among the laboratories and then compared to historical data from various sources. The testing performers involved were Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Naval Surface Warfare Center, Indian Head Division (NSWC IHD), Sandia National Laboratories (SNL), and Air Force Research Laboratory, Tyndall AFB (AFRL/RXQL). These tests were conducted as a proficiency study in order to establish some consistency in test protocols, procedures, and experiments and to compare results when these testing variables cannot be made consistent.

Keywords: Small-scale safety testing, proficiency test, impact-, friction-, spark discharge-, thermal testing, round-robin test, safety testing protocols, HME, RDX, potassium perchlorate, potassium chlorate, sodium chlorate, sugar, dodecane, PETN, carbon, hydrogen peroxide, flour, cumin, glycerin, nitromethane, ammonium nitrate, gunpowder, urea nitrate, sulfur, aluminum, HMX.

IDCA Explosives Safety Testing of Homemade Explosives
Integrated Data Collection Analysis Program

IDCA Program Analysis Report 023 (2012) November 26, 2012
LLNL-TR-604252 (688152)

1 INTRODUCTION

The IDCA was tasked to collect Small-Scale Safety and Thermal (SSST) test data on selected improvised or Home Made Explosives (HMEs) and to evaluate the effectiveness of standard SSST methods for developing safe handling practices for HMEs. The IDCA found several issues when testing HMEs that normally are not observed when testing conventional explosives. These issues can lead to the development of handling practices that may not be accurate assessments of the danger in handling HMEs. The implication of these results was the main topic of the IDCA Final Review in Washington DC at DHS headquarters, September 12, 2012.

All the IDCA principal investigators from each of the participating laboratories attended the review. Mary M. Sandstrom (msandstrom@lanl.gov) and Geoffrey W. Brown (GeoffB@lanl.gov) represented LANL. Mary joined the IDCA shortly after the program began and Geoff joined several months later. Kirstin F. Warner (kirstin.warner@navy.mil) represented the NSWC-IHD. Kirstin was one of the founding principals of the IDCA. Timothy J. Shelley (tim.shelley@tyndall.af.mil) and Jose A. Reyes (jose.reyes.12.ctr@us.af.mil) represented the AFRL, Tyndall AFB. Tim was one of the pre-founding principals, and Jose joined sometime after the program started. Jason J. Phillips (jjphil@sandia.gov) represented SNL. Jason joined the program in the last year. Peter C. Hsu (hsu7@llnl.gov) and John G. Reynolds (reynolds3@llnl.gov) represented LLNL. Peter was one of the original principals of the IDCA. John, along with Becky Olinger (bstreet@lanl.gov) of LANL, started the IDCA concept. Also in attendance was Daniel N. Sorensen (daniel.n.sorensen@navy.mil) of NSWC-IHD, Laura J. Parker (laura.parker@hq.dhs.gov), Program Manager of DHS S&T and Greg F. Struba (greg.struba@associates.hq.dhs.gov), SETA support for DHS.

This report summarizes the presentations and topics discussed at the Final Review. The presentations are attached at the end of this summary and are referred to throughout the summary by page numbers.

2 MEETING PRESENTATIONS

2.1 Schedule

Page 33 shows the schedule for the meeting, lists the topics discussed and the presenters. The meeting started with a summary of the program over the period of performance. The subsequent presentations were about specific technical topics that arose during the period of performance. The meeting was completed with a discussion of the future of the program.

2.2 IDCA Final Review

Because the sponsor had been briefed previously on the structure of the IDCA Program, the traditional review of the objectives of the program and of the Proficiency Test were not presented. Instead, the summary presentation started with a listing of sponsor-driven deliverables during the program followed by which were successfully accomplished and which were not. The following is a summary of these points. The full review of the program is in "The Integrated Data Collection Analysis (IDCA) Program—Final Review," by John G. Reynolds. The presentation can be found on pages 34 to 55.

Collect SSST testing data on HMEs and relevant military standards. This was the primary objective of the IDCA from the onset. To address this, the IDCA conducted SSST testing through a Proficiency Test of 19 HMEs and military explosives (22 sets of materials tested, completion—100 % LANL, 100 % LLNL, 91 % IHD, 32 % AFRL, 9 % SNL); developed the synthesis of MEKP and methyl nitrate (eventually dropped from the program due to safety concerns); and participated in the efforts to organize an International Round Robin for HMEs by TSWG (effort never came to fruition).

Variation in the testing parameters to understand how to test HMEs. The Proficiency test was constructed to vary parameters that might affect testing of HMEs. The following are the sets of tests that interrogate the effect of a specific variable:

- RDX Standard 4 times to set baseline;
- 2 particle size differences in $KClO_3$ to examine the effects of varying particle size of the oxidizer;
- Drop hammer (impact testing) of 10+ materials at 2 or more grit sizes of sandpaper (120-, 150-, and 180-grit) to examine the effects varying the grit size of the sandpaper;
- 2 fuels (sugar and dodecane) with 3 different oxidizers ($KClO_3$, $KClO_4$, $NaClO_3$) and 1 oxidizer ($KClO_4$) with 3 different fuels (Al, C, and dodecane) to examine the effects of different fuel types and structures (solid-solid; solid-liquid);
- 2 different concentrations of H_2O_2 (90 and 70 %) with fuels to examine the effects of varying H_2O_2 concentration;
- H_2O_2 with 4 different organic fuels to examine the effects of varying mixture properties (handling gooey, foamy mixtures);
- 2 component mixture combinations (AN, Gunpowder, AN/Gunpowder; UN/Al and UN/Al/S) to examine the effects of varying solid fuel combinations;
- DSC of 18+ materials with 2 sample holders (vented and sealed) to examine the effects of a closed vs. open DSC system;
- ABL and BAM friction data of 20+ materials to develop a transfer function between the two types of friction measurements;
- Drop hammer data on 20+ materials to compare two analysis methods (Neyer and Bruceton)
- BAM friction20+ materials analyzed by BAM friction by 2 analysis methods (threshold (TIL) and Bruceton (F_{50})) to compare two analysis methods;
- 10+ materials analyzed by 2 ESD methods (ABL and LLNL Custom) to compare performance due to equipment design.

All these sets of experiments probed for issues of concern about applying standard test methods to HMEs.

Statistical variation in SSST testing. Statistical analysis is important in the Proficiency Test to determine statistical significance of data taken among participating laboratories as well as determine statistical significance of data taken within a laboratory. Sufficient test data were available from the Proficiency Test to compare results:

- Among participating laboratories
    - 20+ data sets with at least three participating laboratories for each test;
    - Impact, BAM and ABL Friction, ABL and LLNL Custom ESD were analyzed by accepted statistical methods;
    - DSC Enthalpy data also analyzed for RDX and PETN.
- Within a specific laboratory
    - RDX was tested 4 times by LANL and LLNL; 2 times by IHD;
    - PETN was tested throughout the program by LANL.

Develop and evaluate standard SSST testing methods for application to HMEs. Additional tasking for the IDCA was to assess whether standard safety testing methods are appropriate to use on HMEs. To begin to perform this task, the IDCA eliminated sources of variability in the testing by distributing the test materials from the same batch to all the participants and developing special IDCA procedures for drying, sample preparation and materials compatibility. By standardizing these parts of testing allowed the IDCA to evaluate testing results as a function of how the HME responds when using standard test methods.

The technical presentations below address some of the issues arising when the standard methods are applied to HME SSST testing. These issues are summarized in the conclusions section.

Create and populate a SSST testing data guide. Another of the original tasks for the IDCA was to conduct testing of HMEs and collect the testing results into a compendium to be distributed to all those who are working on HMEs under DHS funding and guidance. The distribution was to extend to the International Community when the test data guide was fully functional. To this end, the IDCA did not complete this effort. However, the task did receive some attention. A beta copy was delivered to Sponsor in 2009 as hard copy and e-file (LLNL and LANL data only on HP/fuels and UN/fuels). The reviews of this version led to revisions of formatting and scope. Table format was redesigned to include links to SSST testing methods and procedures from each contributor. The content was also revised to include aging studies and additional information on hazards. This was the limit of the effort on this task. Unresolved is the inclusion of IDCA Proficiency Test data and solutions for an interactive platform, and access control.

Deliver comprehensive reports on all findings. A set of deliverables to the sponsor from the Proficiency Test was individual reports on each of the materials tested. The IDCA did not meet all these deliverable and has not completed all the program reports on testing and evaluating. However, there are 21 Analysis Reports that compare SSST data among the participants for each material, summarize results compared to military explosives standards, and compare average Proficiency Test data to other sources. There are 113 Data Reports, which are full SSST testing data reports from each participant for each material and/or supporting materials, such as particle size distribution. There are 10 Presentations to groups outside of the IDCA including 3 to TSWG International HME meeting, 1 to DOE and 6 to outside interests, such as professional societies and working groups. A full listing of titles of the IDCA Program Analysis reports and the IDCA Program Presentations is found at the end of this report.

Document real issues arising from the application of standard testing method to HMEs. The program found many such issues. These issues are the basis of the technical presentations that followed the Final Review presentation and are discussed below.

2.3 Experimental methods

To reduce the number of variables in the SSST testing process, the IDCA developed special methods and procedures for drying and handling materials. In addition, to remove the uncertainty in chemical composition, each laboratory was given the material for testing from the same batch. However, the actual testing procedures could be different because each laboratory has their own internal testing protocols. These testing protocols were carefully tracked in case issues arose that might be traced back to the protocols. Jose A. Reyes presents a full summary of the experimental methods in "The Integrated Data Collection Analysis (IDCA) Program—Experimental Methods." The full presentation is found on pages 56 to 74.

Different types of SSST testing equipment used by the different laboratories. Although SSST testing equipment has not changed much in the last 60 years, each laboratory has different versions of the equipment. Figure 2.3.1 shows the different drop hammer instruments for each of the laboratories.

LANL 1954 AFRL 2009 SNL 2012 LLNL 1956 IHD 1945

Figure 2.3.1 Drop Hammer equipment used in the IDCA Proficiency test

The figure shows a wide range of vintages of drop hammers. The very old versions were custom built, and the new versions were purchased. They all work essentially the same but differ in sophistication of controls and flexibility during measurement. This captures much of the variability in SSST testing equipment used by the IDCA—depending upon the year built, the basic designs are the same, but peripherals are different.

How each laboratory conducts a specific test and how they differ from the other laboratories. The first HME tested by the IDCA was the $KClO_3$/sugar mixture. The data showed that the impact testing results differed among the laboratories. Ultimately, the reason for the differences was attributed to sandpapers of different grit size used to hold the sample in place. The IDCA changed procedures and standardized the sandpaper because of this observation. This exercise highlighted the need to know exactly how each participant conducts testing and the need to document any testing method changes. The details of the testing methods and how they changed throughout the period of performance are recorded in the *IDCA Program Analysis Report* **009** and in this presentation.

Changes in testing protocols. As described in the section above, the IDCA did normalize testing protocols to some extent during the Proficiency Test. The main changes were sandpaper grit size in impact testing, sample preparation in impact testing, liquid testing methods and standards, and testing equipment.

Documented procedures. For the Proficiency Test, the IDCA standardized mixing procedures (*IDCA Program Analysis Report* **002**), drying procedures (*IDCA Program Analysis Report* **004**), analysis procedures (*IDCA Program Analysis Report* **001**), and sample preparation procedures (*IDCA Program Analysis Report* **002**). Methods and changes in methods (*IDCA Program Analysis Report* **009**) were also documented throughout the performance period. These standardization procedures along with distributing each material from the same batch to each participant gave the IDCA more confidence that some of the variables normally encountered in SSST testing were eliminated.

2.4 Comparison of Bruceton and Neyer Analysis Methods

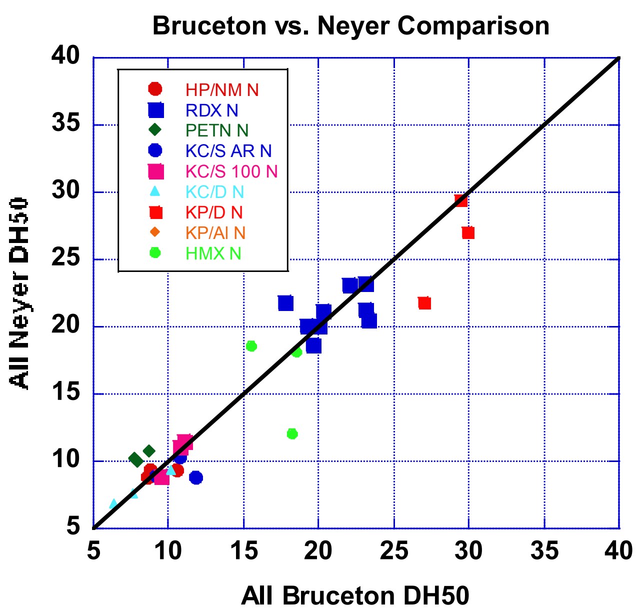
The IDCA employed three analysis methods for impact, friction and spark—modified Bruceton, Neyer and Threshold Initiation Level (TIL). These methods were used as the following:

- Impact—modified Bruceton and Neyer;
- Friction—modified Bruceton and TIL;
- Spark—TIL.

The modified Bruceton method was commonly used because it is fairly easy to apply. One can either calculate the values by hand or quickly write a spreadsheet (that can be shared with others). The TIL method is even easier because it is just an application of an experimental protocol. The Neyer method is also easy to apply but requires purchase of software.

LANL was the only participant to apply both the modified Bruceton and the Neyer method for impact testing on all the materials studied by the IDCA. This provided the data to do a statistical evaluation of the two different analysis methods. Geoffrey W. Brown presents a full analysis of these methods in "The Integrated Data Collection Analysis (IDCA) Program—Comparing Bruceton and Neyer Methods for Determining 50% Reaction Levels." The presentation is found on pages 75 to 93.

This presentation reviews some of the assumptions that go into safety testing of explosives. It also goes through explanation of SSST testing terms, defining the 50% probability of reaction concept and how to experimentally to attain this. The presentation also compares in detail, the two test methods, modified Bruceton and Neyer.



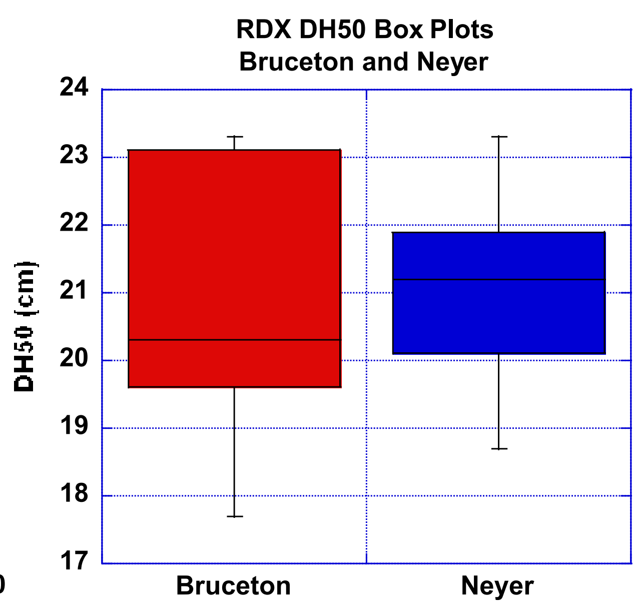


Figure 2.4.1. Comparison of DH_{50} values calculated by the modified Bruceton and Neyer analysis for IDCA Materials—all materials on the left side and RDX on the right side.

Statistical comparison of modified Bruceton and Neyer methods. Figure 2.4.1 shows the DH_{50} (50% probability of reaction) levels as calculated by the Bruceton and Neyer methods of all the IDCA materials using 180-grit sandpaper in the testing. The graph on the left side of the figure shows a fairly close to linear correlation between the modified Bruceton results and the Neyer results. The only exception appears to be PETN, although with only 3 evaluations, and given the proximity of the results to the line, the statistical significance of the apparent deviation is difficult to assess. The right side of the figure contains RDX box plots also showing the similarity of the results from the two methods. ANOVA analysis indicates there are no statistical differences between the two methods in this RDX analysis. The only difference is perhaps the estimation of standard deviation. The Neyer software does a better job of this because it attempts to test at the ± σ points of the distribution.

Bruceton Simulator. The Bruceton simulator can check to see if the parameters selecting test conditions are optimal for the modified Bruceton analysis. Figure 2.4.2 shows simulator results using two different spacing levels in the drop hammer experiment—linear and log.

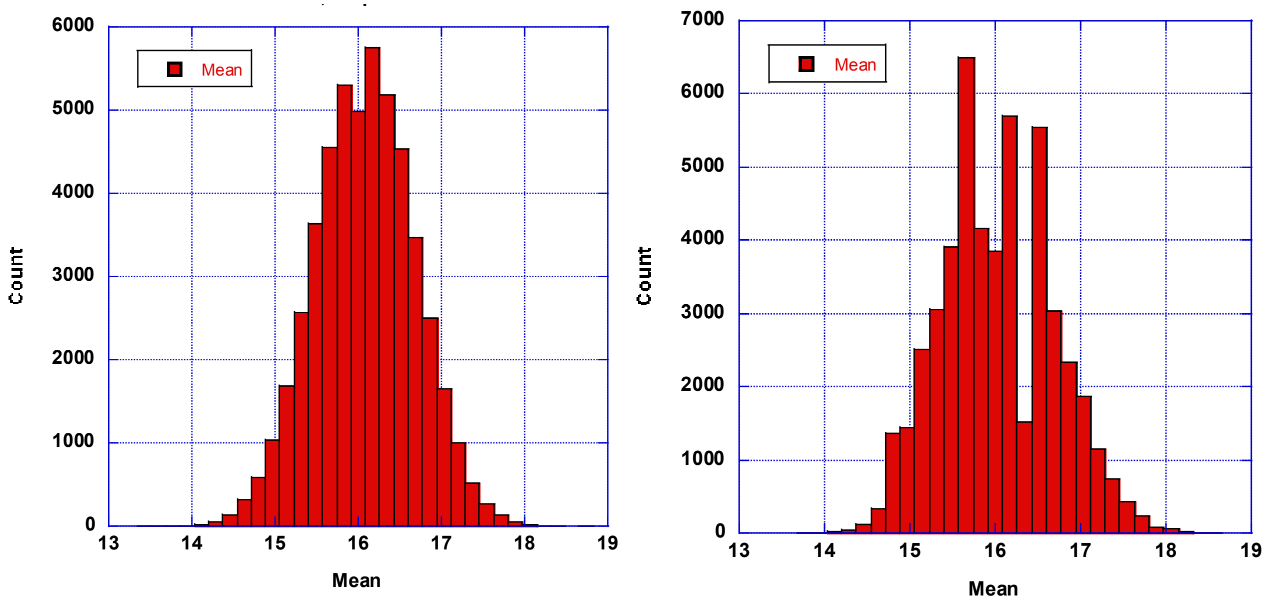


Figure 2.4.2. Simulation of modified Bruceton results comparing linear (left) and log (right) spacing selections.

The input parameters for both simulations are: 50,000 Bruceton evaluations with 25 drops each and the mean equal to 16 cm. In the left side graph, the response is with linearly spaced steps (sigma = 2). In the right side graph, the response is with log spaced steps (sigma = 0.05). The conclusion from the two simulations is for the right side graph; more results come out in wings of histogram. There is a 14% chance that the evaluated mean from 1 test will be more than 1 step away from 16 cm. For the left side graph, there is a 8% chance that evaluated mean from 1 test will be more than 1 step away from 16 cm.

Conclusions of using modified Bruceton and Neyer analysis methods. Bruceton and Neyer D-optimal methods work from the same basic approach and both estimate the mean accurately. Bruceton has to be used correctly: Step size between 0.5 and 2 sigma. Simulation shows a better chance of the result being close to the mean if Bruceton always uses linearly spaced steps. Neyer does a much better job determining sigma (much more useful for comparing results among the participants Neyer does produce good estimates with fewer tests, saving time and expenses. Capital cost of software (< $3000) is likely recovered very quickly. Use of commercial package eliminates errors in spreadsheet functions for homemade Bruceton evaluators.

2.5 Sandpaper Affects HME Impact Test Results

The drop hammer experiment, used for obtaining impact data, can utilize sandpaper to hold the sample in place during the testing. A 2.5-kg weight is dropped from a specified height and this height is used to parameterize the reactivity of the material. The force of the insult is applied to a striker weight that rests on the sample. This impulse is then transferred to the striker weight, which is then transferred to the sample. The sandpaper keeps the sample from spreading during the testing but also provides pressure localization and friction that can cause reaction. During the Proficiency Test, the type of sandpaper used in this test was recognized as a major issue. Timothy J. Shelley presents a full discussion of the sandpaper issues in "The Integrated Data Collection Analysis (IDCA) Program—Sandpaper Affects HME Impact Test Results." The presentation is found on pages 94 to 113.

The first HME that the IDCA tested was $KClO_3$/sugar. When the results were compared among the participants, the DH_{50} for impact testing exhibited some variation among the participants. Table 2.5.1 shows these results (from *IDCA Program Analysis Report* **007**). The average DH_{50} values for the $KClO_3$/sugar, in cm, are: LLNL, 14.9 ± 1.1; LANL, 14.0 ± 3.9; IHD, 14.3 ± 0.6.

Table 2.5.1. Impact Testing results of $KClO_3$/sugar from the IDCA Proficiency Test

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LLNL (120)	1/22/10	23.3	21	14.5	2.18	0.065
LLNL (120)	2/25/10	22.8	28	14.0	0.71	0.022
LLNL (120)	2/16/10	22.8	28	16.1	0.74	0.020
LANL (150)	2/8/10	21.2	13.5	15.5	2.73	0.076
LANL (150)	2/9/10	21.1	14.2	17.7	1.80	0.044
LANL (150)	2/10/10	21.8	13.5	18.8	1.52	0.035
LANL (180)	4/28/10	22.3	<10	10.7	1.88	0.076
LANL (180)	4/29/10	22.1	<10	11.8	4.07	0.147
LANL (180)	5/4/10	22.0	<10	9.2	1.32	0.062
IHD (180)	1/21/10	26	40	14	2.3	0.07
IHD (180)	2/3/10	27	40	15	6.4	0.18
IHD (180)	2/3/10	27	40	14	4.6	0.14

1. Number in parentheses indicates grit size of sandpaper; 2. Relative humidity; 3. DH_{50}, in cm, is by a modified Bruceton method, load for 50% reaction; 4. Standard deviation.

The first dramatic appearance of the sandpaper-dependent results was with the RDX standard. LLNL used 120-grit sandpaper, LANL used 180-grit sandpaper, and both IHD and AFRL used 180-grit sandpaper. Average values for RDX, in cm, were: LLNL, 24.1 ± 0.1; LANL, 25.4 ± 1.3; IHD, 19.3 ± 1.9; AFRL, 15.3 ± 2.3. Ultimately, with other mixtures, such as $KClO_4$/Al, the DH_{50} values exhibited dramatic differences that were attributed to the sandpaper used in the drop hammer test. The average DH_{50} values for $KClO_4$/Al based on grit size are 120, insensitive; 180, 40.9 ± 15.2 cm (14 determinations).

The IDCA members select the type of sandpaper for impact testing based on the characterization category that they describe the explosive and the criteria that are set forth in their controlling documents. In all cases, for the routine testing that the participants do for in-house projects, the choice of the different sandpapers is justified. So the variability in testing methods, such as different sandpapers in the drop hammer, is to be expected. The choice in sandpaper did seem to have a different effect on impact data for the military standards when compared to $KClO_3$/sugar. For many other HMEs, the IDCA found the DH_{50} values were dramatically affected by the choice of sandpaper, suggesting this testing protocol needed some clarification.

Impact sensitivity non-predictively affected by testing conditions. Figure 2.5.1 shows impact sensitivity testing of selected HMEs under six different experimental conditions. The shown DH_{50} values are set relative to an RDX standard (the DH_{50} of standard is subtracted from the DH_{50} of the material setting the standard to 0). A positive DH_{50} value means the material is more stable than the standard; a negative DH_{50} value means the material is less stable than the standard. The standard is tested under the same conditions at which the sample is tested. Three mixtures were tested with two different sandpapers. The experiments were: **1**. $KClO_4$/Dodecane (120-grit sandpaper); **2**. $KClO_4$/Dodecane (180-grit sandpaper); **3**. $KClO_3$/Dodecane (120-grit sandpaper); **4**. $KClO_3$/Dodecane (180-grit sandpaper); **5**. $KClO_4$/Al (120-grit sandpaper); **6**. $KClO_4$/Al (180-grit sandpaper).

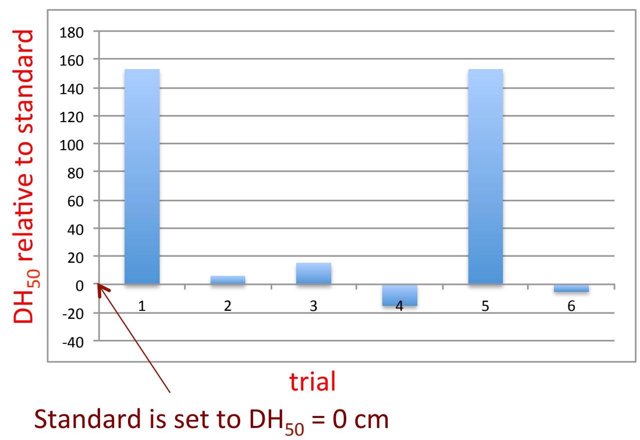


Figure 2.5.1. DH_{50} values of selected mixtures relative to RDX Standard

Figure 2.5.1 shows both mixtures **1** and **2** being less sensitive than the standard, but with **1** being much less sensitive than **2**; mixture **3** being more sensitive to than the standard and **4** being less sensitive than the standard; mixture **5** being much less sensitive than the standard and **6** being slightly more sensitive than the standard. Because the only difference in these mixture pairs is the use of 120- vs. 180-grit sandpaper to hold the sample in place, and *the RDX standard changes in a different way than the mixtures*, no relative or absolute sensitivity assessment of the material is possible, beyond the statement that, under certain conditions, the mixture is observed to be more or less sensitive than RDX. For more details, see *IDCA Program Analysis Report* **022**.

Particle size and sandpaper grit size. One aspect of the sandpaper argument has been the potential mismatch between the size of the grit of the sandpaper and the particle size of the mixture. For the large size grit, very small mixture particles could fall between the grit on the surface of the sandpaper. If so, then the grit is less likely to cause a pressure point for a site of reaction (the striker weight impacts the sandpaper grit, but does not force the solid and the sandpaper into contact with each other).

Figure 2.5.2 shows the relationship between the particle size distribution of two oxidizers, $KClO_3$ and $KClO_4$ and the sandpaper grit sizes. The two different sandpaper particle sizes are shown as the colored rectangles—120-grit (blue) and 180-grit (red). For the $KClO_3$, the particle size overlaps with the grit size of both sandpapers. For $KClO_3$ mixtures, the impact sensitivity is sandpaper dependent, but not very dramatic. In Figure 2.5.1, mixture test pairs **3** ($KClO_3$/dodecane with 120-grit sandpaper) and **4** ($KClO_3$/dodecane with 180-grit sandpaper) are examples of this. However, for $KClO_4$, the particle size range overlaps with only the 180-grit sandpaper (although not much) while the particle size range does not overlap with the 120-grit sandpaper. For $KClO_4$ mixtures, the results from different sandpaper grit sizes are dramatic. In Figure 2.5.1, mixture test pair **1** ($KClO_4$/dodecane with 120-grit sandpaper) and **2** ($KClO_4$/dodecane with 180-grit sandpaper) and pair **5** ($KClO_4$/Al with 120-grit sandpaper) and **6** ($KClO_4$/Al with 180-grit sandpaper) are examples. In these latter cases, the fuel component is liquid in one case and an extremely small particle size solid in the other case, which does not increase the apparent size of the $KClO_4$, so the resulting mixture lies between the grits in sandpaper and just may not be impacted by the striker.

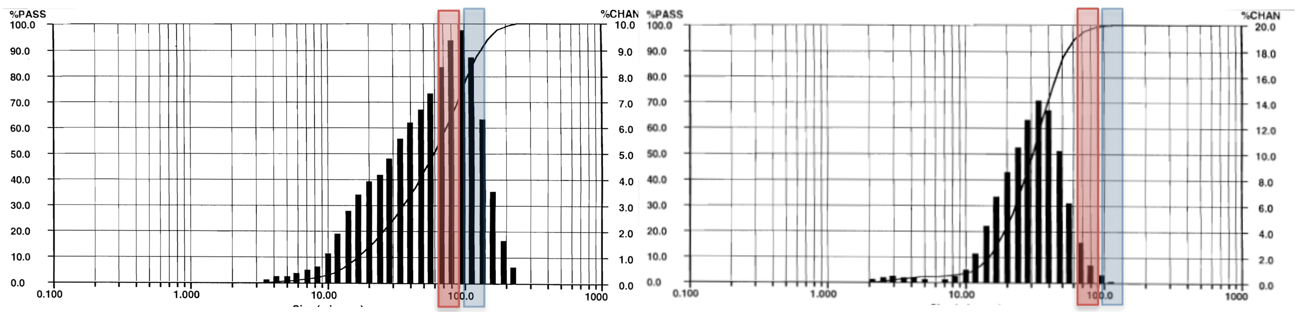


Figure 2.5.2. Particle size distribution of $KClO_3$ (left side) and $KClO_4$ (right side) and 180-grit sandpaper (red overlay) and 120-grit sandpaper (blue overlay). The grit size distributions were estimated form the UAMA (CAMI) specifications for the sandpapers.

Sandpaper has many varied properties. The above is just one facet representing a number of differences in sandpaper. Table 2.5.2 lists the many different properties of the sandpapers used by the IDCA in the Proficiency Test. Any one or more of these properties could account for some of the differences in reactivity expressed when using the different sandpapers.

Table 2.5.2. Sandpaper property comparisons

Property	**120-grit**	**150-grit**	**180-grit**
Average particle size (UAMA), mm	0.115	0.092	0.082
Particle composition	Silicon Carbide	Garnet	Garnet
Surface coverage, actual, particles/mm^2	51	57	54
Surface coverage, calculated, particles/mm^2	59	115	142
Actual surface coverage, %	85	50	38
Volume of particles, actual, mm^3	0.0765	0.0456	0.0324
Volume of sandpaper, calculated, mm^3	0.115	0.092	0.082
Actual coverage, %	67	50	40
Sandpaper thickness, mm	0.391	0.249	0.218
Backing thickness, mm	0.276	0.157	0.136
Backing type	Waterproof	Not waterproof	Not waterproof
Adhesive	Resin	Hide	Hide
Coat	Closed	Open	Open

Conclusions for sandpaper issues. On closer inspection of Table 2.5.2, considerable differences exist among sandpapers used in the Proficiency Test. It is only speculation at this time as to which property is the source of the different values in testing. As well, the cause could be a combination of properties. The IDCA cannot answer at this time—more work is needed. Assessing the explosive sensitivity based on the category (primary, booster, or main charge, which determines the type of sandpaper used for testing) yields the following: $KClO_4$/Al appears to be a main charge high explosive when 120-grit sandpaper is used for the test; $KClO_4$/Al appears to be a primary explosive when 180-grit sandpaper is used. Even though the choice of 120- vs. 180-grit sandpaper does not matter as much when dealing with commercial/military explosives, grit size can have a huge effect in testing HMEs. To standardize testing for many of the HME mixtures, the IDCA adjusted methods about 1/3 of the way through the Proficiency Test to use 180-grit sandpaper from the same manufacturer and lot #. For selected mixtures, testing with other sandpapers was done in addition to testing with the 180-grit sandpaper. Critical for comparison of results, the grit size and composition must be documented.

2.6 ABL and BAM Friction Comparison

The IDCA measured friction sensitivity using two different types of equipment, the BAM and the ABL friction measuring systems. The BAM method was developed by the German Bundesanstalt für Materialprüfung laboratory and the ABL by the Allegany Ballistics Laboratory. The biggest differences in the two apparatuses are that the BAM uses a ceramic plate that is dragged under ceramic pin and the ABL uses a metal anvil that is moved under a stationary grooved wheel. Also, BAM is measured in weight applied to the pin and ABL is measured with force in psig applied to the anvil.

The IDCA included the methods because both are being used in many testing laboratories. The BAM is also a UN certified test. IHD is the only participant that had both pieces of equipment functional and available during the Proficiency Test and examined all of the mixtures and standards. The IDCA continually was trying to find a translation function between the results of the two tests, with the hope that the results of one test could be directly translated into the results of the other test, so both sets of data could be used interchangeably. Kirstin F. Warner presents a full analysis of these two methods in "The Integrated Data Collection Analysis (IDCA) Program—ABL and BAM Friction Data." The presentation is found pages 114 to 130.

Table 2.6.1. BAM and ABL Threshold Initiation Levels (0/20) for Proficiency Test Materials

Material	**ABL (psig @ 8 fps)**	**Material**	**BAM (kg)**
PETN	7.7	$KClO_3$/sugar (-100)[a]	2.3
$KClO_3$/sugar (-100)[a]	30	$KClO_3$/sugar (AR)[b]	3.2
HMX	45	PETN	4.3
RDX Type II Set 1	74	$NaClO_3$/sugar	4.4
AN/Gunpowder	76.6	H_2O_2/cumin	8.6
RDX Type II Set 2	92	HMX	8.6
$KClO_3$/sugar (AR)[b]	123	H_2O_2/flour	11.4
$KClO_3$/dodecane	135	H_2O_2/glycerol	11.8
UN/Al	217	RDX Type II Set 2	11.8
UN/Al/S	217	AN/Gunpowder	12.2
$NaClO_3$/sugar	225	RDX Type II Set 1	15.5
$KClO_4$/dodecane	350	$KClO_3$/dodecane	16.5
AN (-100)[c]	385	$KClO_4$/dodecane	33
H_2O_2/cumin	> 1000[d]	AN (-100)[c]	36.7
H_2O_2/flour	> 1000[d]	UN/Al	> 36.7[d]
H_2O_2/glycerol	> 1000[d]	UN/Al/S	> 36.7[d]
H_2O_2/nitromethane	> 1000[d]	H_2O_2/nitromethane	> 36.7[d]

a. $KClO_3$ separated through a 100 mesh sieve; b. As received separated through a 40-mesh sieve; c. AN separated through a 100-mesh sieve; d. No reaction at the maximum force that can be applied by the equipment.

The search for a translation function between ABL and BAM friction testing. Overall, 21 data sets comparing BAM and ABL friction results were taken during the Proficiency Test. Not all data sets were complete for various reasons—insensitivity exceeded the limits of the equipment; mixture could not be properly contained for the test; positive reaction (go) could not be reproducibly defined.

Table 2.6.1 shows the Threshold Initiation Level (TIL) values for each of the materials in order of sensitivity as measured by ABL and BAM friction tests. Clearly, there is a difference in order of sensitivity, so the two methods are not assessing the sensitivity of the materials the same. PETN and $KClO_3$/sugar appear to be some of the most sensitive materials in both tests. The H_2O_2 mixtures show no sensitivity in the ABL test, but have various sensitivities in the BAM test. Only the H_2O_2/nitromethane mixture shows no sensitivity in both tests.

Figure 2.6.1 shows the attempt to correlate the two methods. The left graph shows the TIL values for ABL testing vs. the TIL values for BAM testing. The right graph shows the F_{50} values (calculated by a modified Bruceton method) for ABL testing vs. BAM testing. A linear fit was attempted on both data comparisons. Clearly, there is no relationship between the two methods for either for TIL or F_{50} values for when inspecting the residuals (0.472 and 0.1784, respectively). However, when subgroups are analyzed, there may be some correlations (see presentation).

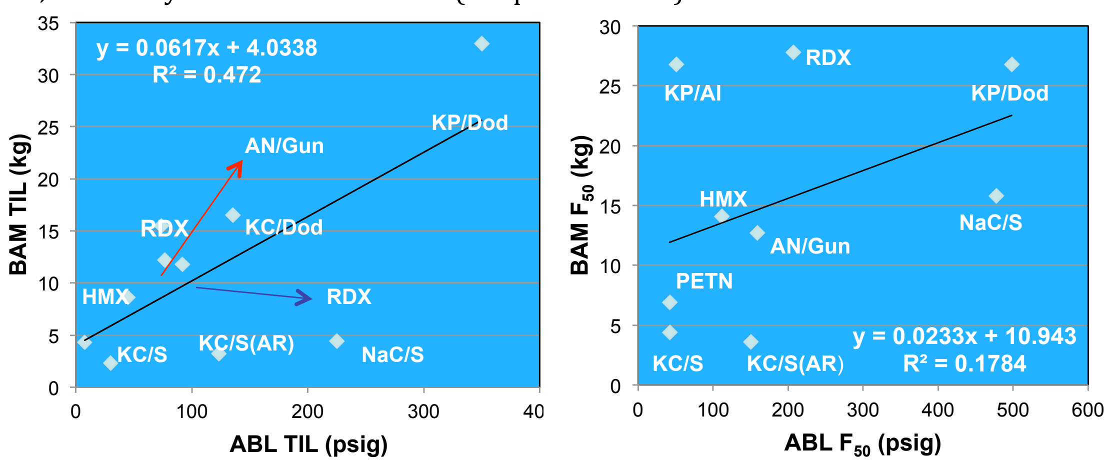


Figure 2.6.1. ABL and BAM TIL and F_{50} friction sensitivity data.

Conclusions from ABL and BAM Friction Testing. This testing effort shows:

- No obvious translation function—order of sensitivities do not intrinsically match;
- Order the sensitivities of some materials is in a similar sequence (comparisons within a structural class may be possible);
- TIL data (no go) correlates better than corresponding F_{50} data;
- The two (2) test methods show that $KClO_3$/Sugar (-100) was the most sensitive HME;
- Liquids/pastes are less sensitive based on ABL data compared to the corresponding BAM data with the exception of H_2O_2/Nitromethane mixture;
- Liquids and pastes need better protocols for testing on ABL.

2.7 ESD Equipment Comparison

For ESD testing, in the beginning of the IDCA Proficiency Test, LANL, IHD and AFRL used ABL equipment and LLNL used a custom-built system. This LLNL system was custom built in the 1970s and was designed with a 510-Ω resistor in the circuit to mimic the human body. However, during the Proficiency Test, LLNL was able to obtain funds to purchase a new ABL ESD, which was brought on-line about ½ way through the testing. At that time, some of the earlier tested mixtures were retested and the results were compared with the custom-built system and the other ABL systems. These other ABL systems are of various vintages. Peter C. Hsu presents a full comparison of these methods in "The Integrated Data Collection Analysis (IDCA) Program—ESD Testing Comparison." The presentation is found on pages 131 to 141.

ABL ESD results compared to the 510-Ω custom built system. Table 2.7.1 compares the results of the ESD testing using the LLNL custom-built system to the ABL systems used by the various participants. For most of the materials listed the LLNL custom-built system indicates insensitive reactivity. Only the $KClO_4$/Al mixture exhibits ESD sensitivity. For the ABL systems, all laboratories reported measureable sensitivity of these same materials.

Prior to enrolling the ABL ESD system purchased by LLNL into the Proficiency Test, comparison of ESD results was difficult between LLNL and the other participants. However, the retested materials by LLNL gave results that were now reasonably comparable to those of the other participants.

Table 2.7.1. Comparison of ESD TIL levels of the LLNL custom-built system and the ABL ESD systems

Sample	**Custom 510-Ω, TIL**	**ABL 0-Ω, TIL**	**ABL, 0-Ω Above TIL**	**Lab**
RDX	0/10 @ 1.0 J	0/10 @ 0.038 J	1/3 @ 0.063 J	LLNL
		0/20 @ 0.025 J	1/3 @ 0.063 J	LANL
		0/20 @ 0.095 J	1/7 @ 0.165 J	IHD
		0/20 @ 0.028 J	1/3 @ 0.063 J	AFRL
HMX	0/10 @ 1.0 J	0/10 @ 0.065 J	1/8 @ 0.075 J	LLNL
		0/20 @ 0.025 J	1/5 @ 0.063 J	LANL
PETN	0/10 @ 1.0 J	0/10 @ 0.031 J	2/5 @ 0.038 J	LLNL
		0/20 @ 0.025 J	1/4 @ 0.063 J	LANL
UN/Al	0/10 @ 1.0 J	0/10 @ 0.038 J	1/10 @ 0.063 J	LLNL
		0/20 @ 0.125 J	1/6 @ 0.25 J	LANL
$KClO_4$/Al	0/10 @ 0.25 J	0/10 @ 0.088 J	2/3 @ 0.013 J	LLNL
		N/A	3/8 @ 0.063 J	LANL
		0/20 @ 0.015 J	1/4 @ 0.023 J	IHD
$KClO_3$/sugar	0/10 @ 1.0 J	NA	NA	LLNL
		0/20 @ 0.063 J	2/3 @ 0.125 J	LANL
		0/20 @ 0.165 J	1/3 @ 0.326 J	IHD

Improved detection of positive ESD events. A difficult part of ESD testing (or any testing for that matter) is proper detection of a positive/negative event (go/no-go). Traditionally, the detection was based on visual observation of some type of reaction over baseline. Baseline for ESD is the action and sound of a spark discharging through a material without causing an energetic reaction. This discharge can be just a spark discharge with a little noise, but also can be a flash or burn. The distinction between the baselines for a specific material and an ESD-driven reaction is difficult and takes much experience to do correctly. It is operator-dependent, and therefore somewhat subjective, casting some doubt on the accuracy of testing results. The field of SSST testing recognizes this problem and is trying to develop measurement equipment that will take the operator subjectivity out of the equation.

The spark in the ESD test, when it interacts with organic based materials (such as $KClO_3$/sugar), produces CO_2, CO and sometime NO_x. These are defined, volatile gases that are lightweight and can be detected by various types of meters. If these gases are monitored, detection can be shifted from observational to instrumental, increasing the credibility of the data collection, assuming the instrumentation is used correctly.

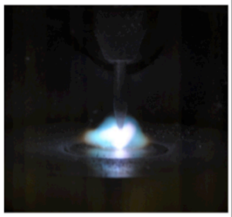
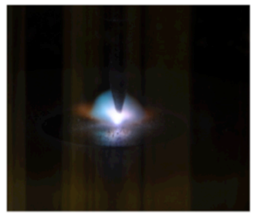
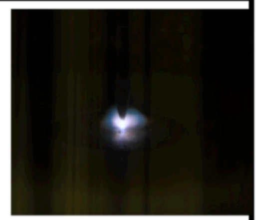
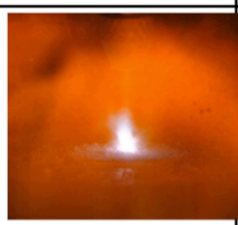
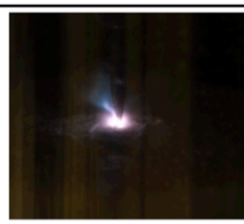
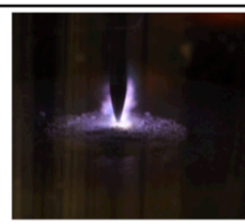
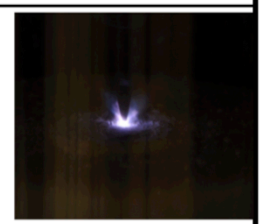
Capacitance Level (μF)	0.1		0.02				0.012	
Blank Image								
Test Image								
Gas	CO_2	CO	CO_2	CO	CO_2	CO	CO_2	CO
Starting Conc. (ppm)	384	90.5	344	81.5	344	86.0	364	91.0
Ending Conc. (ppm)	1100	157	351	89	403	99.3	371	94.9
Δ Conc. (ppm)	**716**	66.5	7	7.5	**59**	13.3	7	3.9
Result	**GO**		**NO-GO**		**GO**		**NO-GO**	

Figure 2.7.1. ESD testing of PETN monitoring visible emissions and gas evolution.

Figure 2.7.1 shows some of the difficulties in ESD detection using PETN as an example. Comparing the blank and test image at 0.1-μF capacitance level indicates the test image clearly shows a reaction, with a much more intense flash. The gas concentration data before and after testing corroborates that the ESD spark caused a reaction. Both CO_2 and CO increased greatly over baseline. This clearly demonstrates the technique. Comparing the blank and the test image at the 0.012–μF capacitance level indicates no difference and visually there would be no reaction assigned, although there is a flash. The gas data supports this also. However, the real advantage of having the gas detection comes at the transition point between go/no-go. At the 0.02-μF capacitance level, there are two examples. Visually, it would be only the most experienced operator that could tell the difference between the tests and the blank—very little visual difference. However, the gas analysis gives supporting information for the differentiation between go and no-go.

Conclusions from the ESD testing comparisons. The inclusion of the new ABL ESD testing equipment by LLNL in the Proficiency Test added extra information to the testing results. This led to better comparisons of spark sensitivity. The inclusion of detection equipment into the ESD go/no-go process also shows promise in making more accurate determinations particularly when organic materials are involved.

2.8 Outstanding Thermal Issues

In the Proficiency Test, results show the application of standard thermal analysis methods used on conventional explosives does not always give clear results when applied to HMEs. Standard methods are: constant heating rate (IDCA used 10°C/min), open pinhole lid on the sample holder, and < 3 mg sample size.

Several of the HMEs studied proved to have thermal behavior that was not initially reproducible, so further examination was required. Mary M. Sandstrom presents the full listing of the results of these studies in "The Integrated Data Collection Analysis (IDCA) Program—Thermal Studies: Issues in application of standard safety test methods to Homemade Explosives." The presentation is found on pages 142 to 185. Specific examples are discussed: 1) Standards RDX and PETN show how standard methods are applicable to military and tradition explosives; 2) $KClO_3$/sugar mixtures fall victim to sampling issues; 3) $NaClO_3$/sugar mixtures are also subject to sampling issues; 4) $KClO_3$/dodecane mixtures suffer from a fuel volatility issue; 5) $KClO_4$/dodecane mixtures suffer from the same fuel volatility issue; 6) H_2O_2/fuel mixtures lack definition because of oxidizer volatility issues.

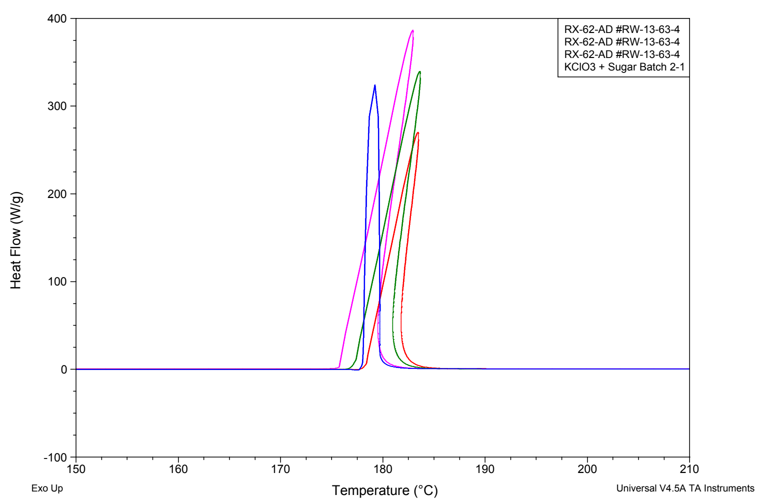


Figure 2.8.1. $KClO_3$/sugar DSC at 10°C/min heating rate, large sample size.

Energetic material overdriving DSC performance. One of the most illustrative materials demonstrating the failure of standard DSC test methods to evaluate HME thermal properties is the $KClO_3$/sugar mixture. Figure 2.8.1 shows a DSC profile of this mixture under standard operating conditions. The exothermic feature has a maximum of around 180°C. It also has an abnormal shape—narrow but slanted. This is a machine artifact due to too much energy release over a very short period of time. In a sense, the sample is over driving the heating, so the DSC heating shuts down for a short time. The solution to this is to use a much smaller sample. Any negative slope on the front of the exothermic feature or positive slope on the backside of the exothermic feature indicate the sample size is too large. The correct sample size may only be determined by previous results or trial and error.

Representative sampling issues. Reducing the sample size because of the overdriving effect above causes another type of problem—obtaining an representative sample. Figure 2.8.2 shows the DSC data for the stoichiometric $KClO_3$/sugar mixture under identical conditions as used in Figure 2.8.1, but this time at a much smaller sample size. Three exothermic features are visible—at ~ 180°C which is assigned as the $KClO_3$/Sugar mixture reacting (sugar melts and then mixes), at ~ 220°C which is assigned as the sugar carbonizing (sugar that did not react), at ~ 340°C which is assigned as the $KClO_3$ melting and reacting with residual carbon. This is a stoichiometric sample, so the 180°C exothermic feature should be the only feature observed.

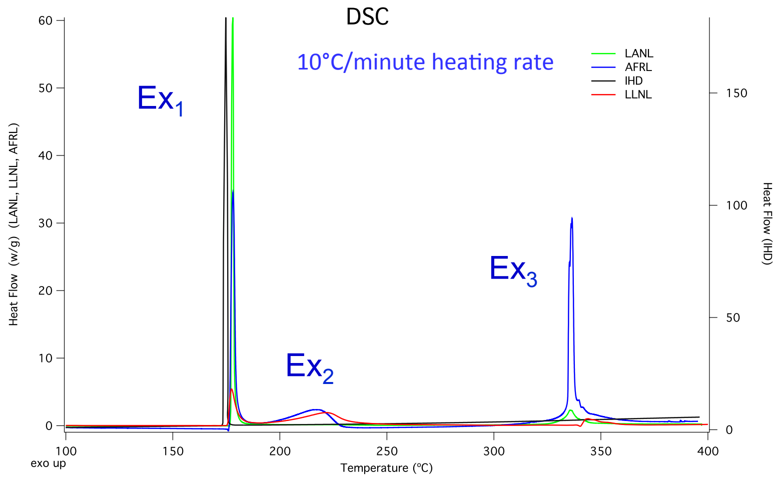


Figure 2.8.2. $KClO_3$/sugar DSC at 10°C/min heating rate, small sample size.

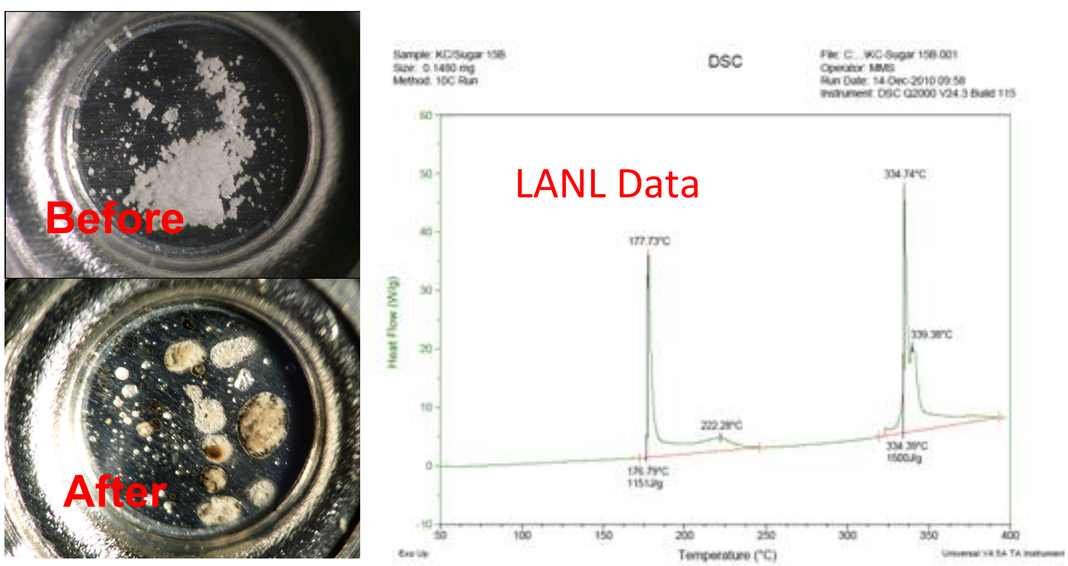


Figure 2.8.3. Photographs and DSC of $KClO_3$/sugar mixture, 0.15 mg sample before and after heating at 10°C/min.

Figure 2.8.3 shows possibly why multiple exothermic features are observed. A single batch of sieved $KClO_3$/sugar was prepared according the IDCA mixing protocols. Standard DSC conditions were used except for sample size—hermetically sealed Al sample holders with 70-μm pinhole lids; TA Instruments Q2000 DSC; ramp rate 10°C/min. Duplicate sample holders mwere loaded with 0.05 mg, 0.10 mg, 0.15 mg, 0.20 mg, 0.25 mg and 0.30 mg samples and one duplicate was run from 40°C to 250°C (sugar melt regime) and the other to 400°C ($KClO_3$ melting regime). Pictures were taken of each sample before it was sealed. The samples were run up to either 250°C or 400°C. The pans were reopened and pictures were taken of the residue left in the pan. These results were then compared to the DSC traces.

The 0.25 mg sample exhibited the single over driven exothermic feature, as seen in Figure 2.8.1. However, the smaller samples exhibited multiple features as seen in Figure 2.8.2. The speculated cause is seen in Figure 2.8.3, which is a photograph of the DSC sample holder before and after heating for the 0.15 mg sample. In the photograph of the before case, clearly the sample does not cover the entire pan. The empty space in the pan may allow the material to segregate during subsequent handling, leading to regions that are locally either fuel rich or oxidizer rich instead of stoichiometric. This may even produce regions that consist of only the fuel or oxidizer alone. These localized regions have different mixture ratios which could lead to the multiple exothermic features seen in Figure 2.8.2. Also borne out in the photograph of the sample pan after heating, are regions of different color are observed, indicating different residue and by extension, the uneven distribution of reactants affecting the chemistry.

Conclusions from DSC studies of HMEs. In most HME cases, the IDCA found issues with applying standard DSC analysis methods. Obtaining a representative sample is a significant issue, whether for mixing two solids where uneven distribution can easily occur at the very small sample size, or mixing a solid and a liquid, where volatilization of the liquid fuel prevents contact with the solid. Other issues such as oxidizer volatility and pretreatment conditions can also play into the problem. The recommendation at this point is to work with these materials on a case-by-case basis, and to not blindly apply standard methods.

2.9 Effect of Pan Type on Ammonium Nitrate Decompositions

Ammonium nitrate (AN) and AN/Gunpowder mixture were tested by LLNL, LANL, IHD, and AFRL. The most notable part of the testing was that the results for the AN were as inconsistent as any of the materials studied in the Proficiency Test. Although all aspects of the testing—impact, friction, ESD and thermal—had issues, the DSC results were particularly confusing. The testing results were further examined to determine the cause of the confusion. Daniel N. Sorensen presents the full results of this study in "The Integrated Data Collection Analysis (IDCA) Program—Effect of Pan Type on Ammonium Nitrate Decompositions." The presentation is found on pages 186 to 194.

The participants all had varied results for the thermal decomposition of AN. Temperature ranges of endothermic features and enthalpy values were different (LANL and IHD enthalpy values were about 1/3 of the LLNL values). There was also a disagreement between DSC observation and intuition because the region where oxidizer was decomposing was exhibiting endothermic decomposition where exothermic decomposition is expected. As well, the literature shows this same disagreement—Gunawan vs. Oxley.

Figure 2.9.1 exhibits the DSC profiles of AN in the literature. The left profile is from Gunawan et al. and the right profile is from Oxley et al. The profiles are similar except for exact minimum temperature of the endothermic features and the high temperature transition is an endothermic feature in Gunawan is an exothermic feature in Oxley. The former issue can be explained by the different heating rates. An exothermic feature is expected for the latter issue because the feature is due to an energetic material decomposing.

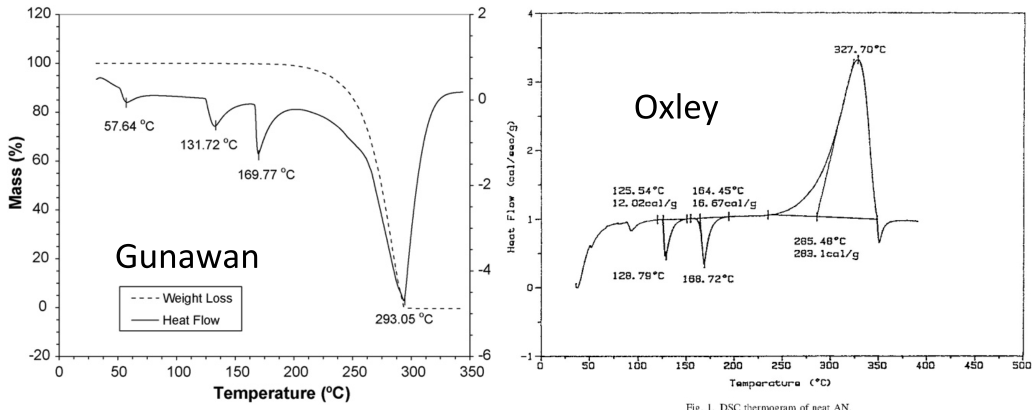


Figure 2.9.1. DSC profiles of AN by Gunawan and by Oxley.

The differences in the high temperature features are simply explained by the type of DSC sample cell that is used for the measurement. The pinhole vented sample holders (standard type used by the IDCA) allow for the gases to escape causing evaporative cooling, which overrides any positive heat flow because of the mass of the sample cell. When the gases are not allowed to escape, an exothermic feature is observed instead. Figure 2.9.2 exhibits this behavior from the IDCA participants.

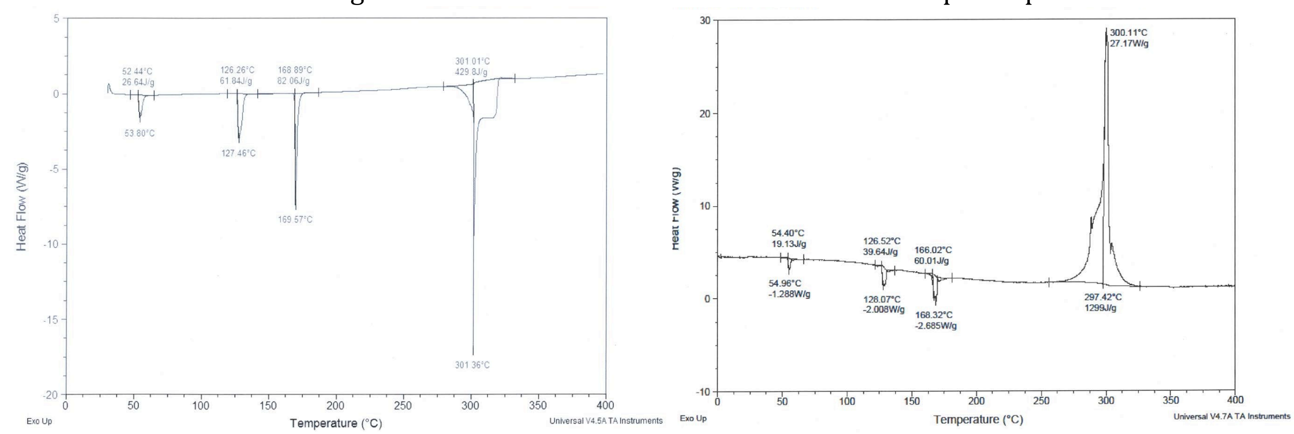


Figure 2.9.2. DSC of AN using a pinhole sample cell (left) and gold sealed cell (right) at 10°C/min heating rate.

The left side of Figure 2.9.2 shows the AN sample heated in the standard Proficiency Test DSC sample holder with a pinhole sample lid. The right side of Figure 2.9.2 shows the AN sample heated in a gold sealed sample holder (Gold High Pressure pans, SWISSI crucibles sold in US by Fauske). The high temperature exothermic feature is clearly seen.

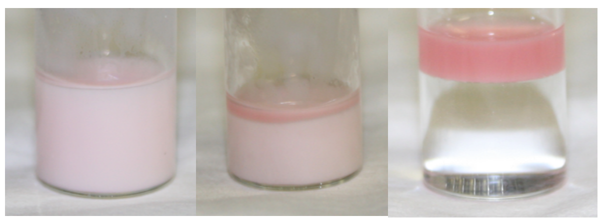
Conclusions from AN DSC experiments. The thermal behavior of AN in the open vs. sealed sample holder aligns with the thermal behavior of other HMEs. In this case, the sealed sample holder (sealed to high pressure) was the solution. This is not always the solution, as in the case of $KClO_3$ and $KClO_4$ mixtures with dodecane where the sealed system gave some indication of the exothermic thermal behavior, but not a complete assessment of the enthalpy. The complete solution may be the need to utilize alternate methods of characterizing thermal behavior of HMEs.

2.10 HME Aging Studies

Some HME mixtures have been known to change upon aging. The thermal runaway of H_2O_2/fuel mixtures is fairly well noted in the HME testing community. One aspect that is not well appreciated is how aging affects SSST testing. Because the IDCA included some of the H_2O_2/fuel mixtures in the list of HMEs, a limited study on aging of these mixtures was performed. Peter C. Hsu presents the results of this study in "The Integrated Data Collection Analysis (IDCA) Program—Aging Studies." The presentation is found on pages 195 to 203.

Although aging was not directly called out in the original test matrix, some aging studies were performed along the way to better bracket the temporal aspects of HME safety testing—when to sample, how long there is enough stability to establish response to insult, and when the material is not safe to even test. The stability of the HME varies depending upon the mixture. Because some of the instability is autocatalytic, the sample size makes a difference. As well, because these mixtures tend to undergo chemical reactions from the onset, time from mixing is a critical test parameter.

Some HME mixtures clearly show that chemistry is occurring with time. Figure 2.10.1 shows an example of H_2O_2 mixed with diesel fuel. The mixture after 0.5 hour is homogeneous and lightly pink; after 5-days is beginning to form two phases; and after 11 days clearly has two phases, one colored and one colorless.



0.5 hours 5 days 11 days

Figure 2.10.1. Photographs of H_2O_2/diesel fuel over a period of 11 days.

The relevant question is how this affects the SSST testing data. To address this, selected H_2O_2/fuel mixtures were tested over a period of several days by SSST testing methods and calorimetry.

Temporal aspects of impact sensitivity of H_2O_2/fuel mixtures. Table 2.10.1 shows the impact sensitivity of selected H_2O_2/fuel mixtures over several days. The H_2O_2/nitromethane mixture appears to change slightly in sensitivity over a period of 3 weeks. The H_2O_2/cumin mixture appears to become less sensitive after 1 week (experiment was ended) and the H_2O_2/drink mix becomes insensitive within 2 weeks.

Table 2.10.1. DH_{50} values for selected H_2O_2/fuel mixtures over 3 weeks aging

Mixture[a, b]	**2 hours**	**3 days**	**1 week**	**2 weeks**	**3 weeks**	**Observations**
H_2O_2/NM	30 cm	35 cm	34 cm	nm	32 cm	Clear color
H_2O_2/Cumin	42 cm	nm	72 cm	nm	nm	Color change, foam
H_2O_2/Drink mix	56 cm	nm	nm	> 177 cm	nm	Color change, foam Ppt. of fine particles

a. H_2O_2 concentration 90%; b. nm = not measured;

Temporal aspects of heat flow in the H_2O_2/flour mixture. Heat flow calorimetry shows instability over time in these types of mixtures. Table 2.10.2 shows the heat flow for an H_2O_2/flour mixture as a function of time. After about one hour, there is heat flow out of the system, indicating exothermic reactions are proceeding, so the composition of the material is likely changing. After 1 day, this heat flow has dropped significantly. However, after 4 days, the heat flow increases indicating some change in the exothermic behavior. Eventually the heat flow goes to 0 after 16 days indicating the reaction is completed.

Table 2.10.2. Heat Flow for an H_2O_2 (70%)/flour mixture

Aging Time	**Heat Flow, mW/g**
< 1 hour	19.9
1 day	3.0
4 days	4.5
8 days	1.0
16 days	0.0

Conclusions from the aging studies. Although the data in this summary of the presentation use examples that are either become more stable over time or do not change over time, there are examples of materials that become more sensitive over time. The conclusions from these aging studies indicate that with HMEs, because they are chemically reactive materials, the stability must be determined on a case-by-case basis to develop safe handling practices.

2.11 HME Testing Capabilities at SNL

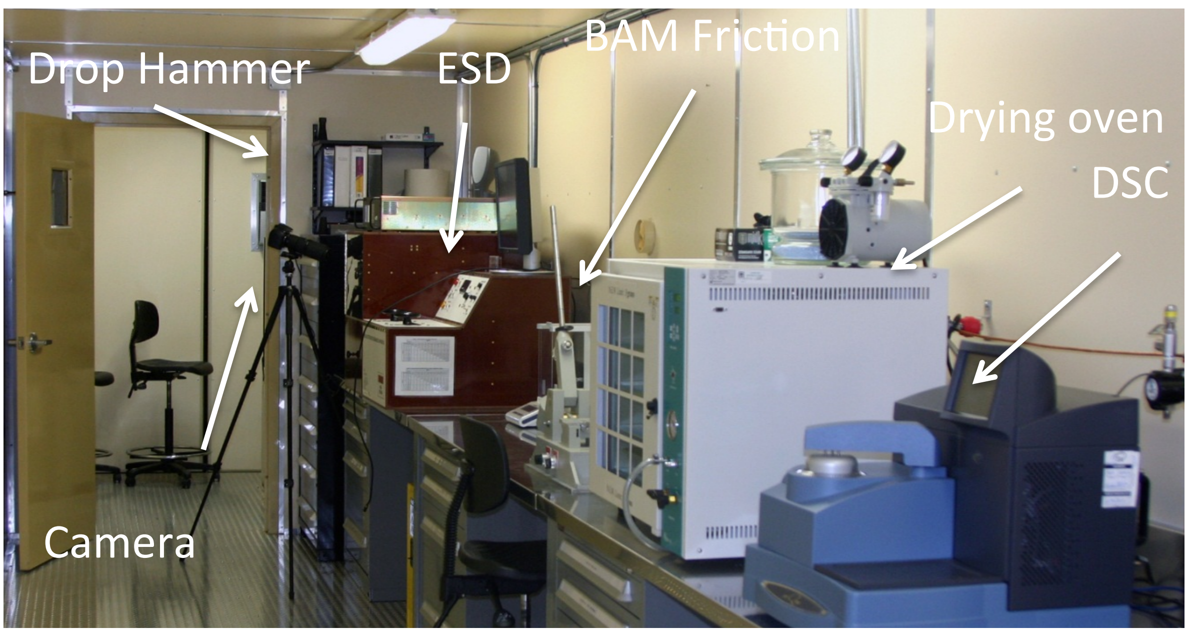


Figure 2.11.1. The new SSST Testing Equipment at SNL.

At the onset of the IDCA Proficiency Test, SNL did not have applicable SSST testing equipment to participate in the data collection on HMEs. As a result, their role was to support characterization efforts of the materials used in the Proficiency Test. Their efforts included scanning electron microscopy analysis and synthesis development as well as other support. During the Proficiency Test, SNL was able to obtain funds to procure the necessary test equipment and staff the operation of that equipment. By the end of the IDCA Program, RDX and PETN were tested and the results were added to the Program Analysis Reports. Jason J. Phillips presents the testing set up in “The Integrated Data Collection Analysis (IDCA) Program—HME Sensitivity Testing Capabilities at Sandia National Laboratories.” The presentation is found on pages 204 to 217.

Figure 2.11.1 shows the equipment configuration. The testing facility has been constructed in transportainers that are climate controlled. The facility has the standard SSST testing equipment—drop hammer, BAM friction, ABL ESD, DSC—but also a camera and gas meters to assist in detection.

Conclusions from the new SNL testing equipment. In addition to having the standard SSST testing equipment, SNL also has a digital single lens reflex camera and gas analyzers to help with the go/no-go observation detection issues.

2.12 Statistical Evaluation

A primary request of the IDCA was to examine the statistical significance of the Proficiency Test results. Enough data was taken by the participants to apply some statistical methods and make various conclusions. These evaluations helped find hidden parameters, determine whether the results are different, and helped focus attention on issues that are method dependent. The participants have measured different results for many of the materials by all the testing methods. These differences lead to many questions such as:

- Are these differences statistically significant or just perceived to be different;
- Are the differences significant for safety; what causes those differences;
- What are the average material properties for the data sets that are not different;
- What are the rankings of these materials; what are the variations in measured sensitivities?

Geoffrey W. Brown presents a full discussion of the statistical issues and analyses in “The Integrated Data Collection Analysis (IDCA) Program—Statistical Analyses Applied to SSST Testing of HMEs and Standards in the IDCA Program.” The presentation is found on pages 218 to 252.

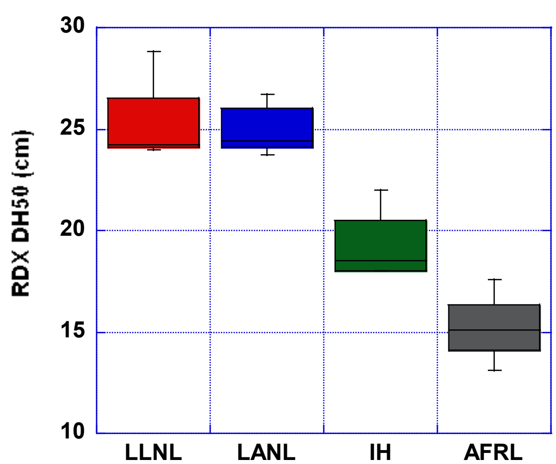


Figure 2.12.1. Box plots of data of RDX measured the first time.

Statistical evaluation of RDX data from LLNL, LANL, IHD, and AFRL. In the Proficiency Test, RDX was the standard and tested in triplicate (or more times) throughout the program. This material was tested first before any of the HMEs, and the results were used as baseline as well as a comparison to document the performance of each laboratory. Figure 2.12.1 shows box plots of the data from the first examination of RDX. In the box plot, the horizontal line is the median, the colored box is 50% of data, and the vertical bars are the maximum and the minimum. The box plot assesses equality of means and variation within and across laboratories. It makes the following assumptions: no outliers for single lab on single grit sandpaper; measured standard deviations are biased, not used for analysis of variance (ANOVA).

From a cursory examination, the LLNL and LANL data appear similar, with the means and variances almost identical. IHD and AFRL data appear different than LLNL and LANL data, as the means are up to 35% lower. ANOVA analysis with assessment of differences by Tukey's method shows that the results can be grouped where LLNL and LANL results are in one group, IHD and AFRL results are the other group and that these two groups are significantly different to the 95% confidence level. Likewise, Fisher analysis of differences divides the results into three groups where IHD and AFRL are statistically different.

Trends in the Proficiency Test data. A useful way to express the large set of data is to show the average values for impact testing (DH_{50} by Bruceton, for example) by a specific laboratory and compare the results to the average values for all the laboratories for that specific material. Figure 2.12.2 shows drop hammer test results for the whole suite of Proficiency Test materials.

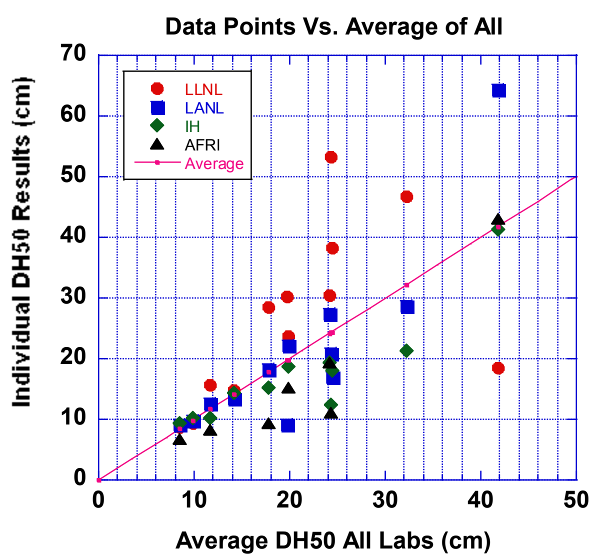


Figure 2.12.2. Comparison of impact data for Proficiency Test materials among laboratories with the average values (DH_{50}, cm).

The graph shows the individual laboratory averages of a specific material (y values) compared to the average for all the laboratories (x values). The analysis shows:

- LLNL (the red dots) has highest values when the DH_{50} value is below 40 cm probably due to differences in microphones (sensitivity and placement) affecting detection and instrument performance at low drop heights;
- LANL (the blue dots) has highest values when the DH_{50} value is above 40 cm due to differences in microphones (sensitivity and placement) affecting detection and instrument performance at high heights (also some anvil configurations);

- AFRL (black triangles) generally has the lowest DH_{50} values when compared to the rest of the participants with the largest striker weight affecting instrument performance, operator sensitivity affecting detection;
- IHD (green diamonds) has DH_{50} values generally below corresponding LANL values due to the effects of operator sensitivity vs. microphones for detection on nearly identical instruments.

Figure 2.12.3 shows a similar graph for BAM friction values. LLNL always has the highest values compared to the other laboratories and the average, which is likely due to the LLNL system being completely enclosed, while the systems of the other participants are open. In addition, LLNL has a HEPA filter system attached. LANL always has values lower than IHD possibly caused by room acoustics, operator differences, and/or humidity.

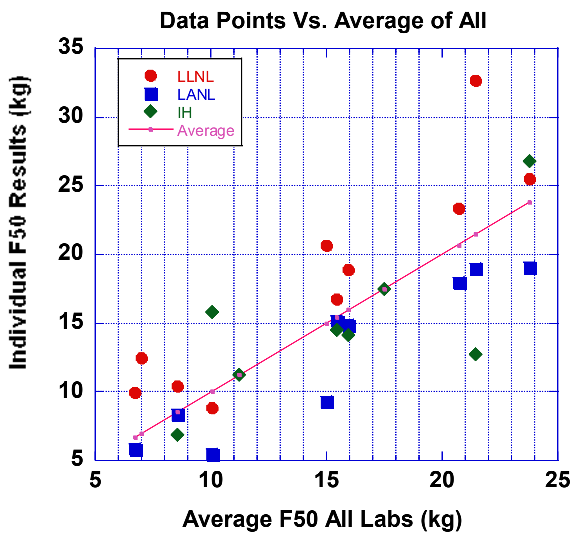


Figure 2.12.3. Comparison of BAM friction data for Proficiency Test materials among laboratories with the average values (F_{50}, kg).

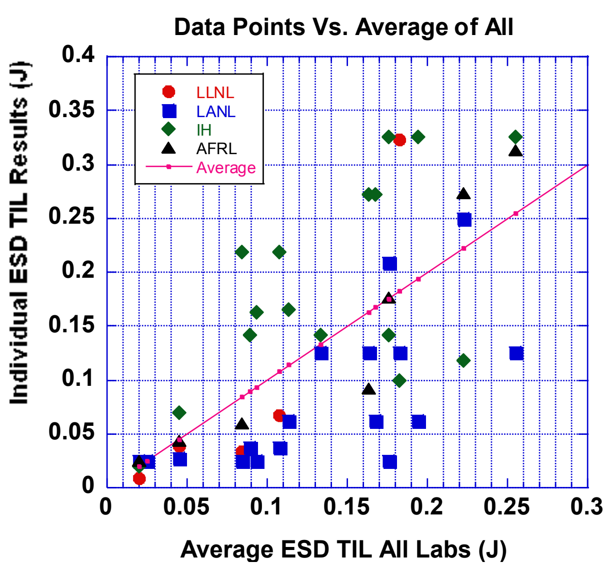


Figure 2.12.4. Comparison of ABL ESD data for Proficiency Test materials among laboratories with the average TIL values.

Figure 2.12.4 shows a similar graph for ABL ESD TIL values. IHD results tend to indicate a more stable material than the LANL results indicate on the corresponding material. This could be due to IHD having an older instrument, different needles, higher humidity, and detection with the room lights on vs. off. LLNL and AFRL have limited data sets with the ABL device.

Conclusions from the statistical analyses. There are many conclusions from this statistical examination. The IDCA laboratories do obtain significantly different results on many materials. The IDCA understands some of the causes, but some will be hard to change. Absolute sensitivity values are not a good cross-lab comparison, while relative sensitivity rankings/order assigned by each lab are better comparisons and are not drastically different.

2.13 IDCA Conclusions from the Proficiency Test

After the technical presentations were complete, conclusions from the IDCA Proficiency Test were scheduled to be presented. However, due to time issues these were not presented. However, the conclusions presentation is included and is shown on pages 253 to 262. Below is a summary of these conclusions.

Solids (essentially pure, not mixtures):
- Standard SSST Testing methods work well;
- Testing parameters, such as sandpaper grit size and particle size must be well documented and carefully followed;
- For some solids, drying is important.

Solid-solid mixtures:
- Obtaining a representative sample is difficult if impossible;
- Sampling is a real problem for DSC at any sample size due to the energetic release;
- Particle size mismatch in the mixture will cause inadequate measurement of sensitivity.

Solid-liquid mixtures:
- Volatile component can escape before testing;
- Particle size mismatch of the solid component with the sandpaper can cause inaccurate measure of sensitivity in impact testing;

Relative sensitivity compared to standards:
- Sandpaper grit size can affect the sensitivity of the standard differently than the mixture;
- The absolute and relative sensitivity of the mixture changes as a function of the sandpaper grit on impact testing;
- This effect is not systematic.

Absolute sensitivity
- Many of the experimental parameters have the potential to cause variation in data when comparing different laboratories;
- Detection by observation varies depending upon the sensory perception of the observer;
- Detection by measurement varies depending upon detection set up. LLNL and LANL use microphones for impact testing (a no-go background is set; a go is decibels above). Other laboratories rely on personal observation;
- Local environments (humidity, temperature) as well as pretreatment may affect sensitivity.

Specific cases
- $KClO_3$/Dodecane changes relative sensitivity to RDX depending upon sandpaper grit size;
- $KClO_4$/Dodecane changes relative sensitivity to RDX depending upon sandpaper grit size;
- $KClO_4$/Al changes relative sensitivity to RDX depending upon sandpaper grit size;

- $KClO_3$/Sugar cannot be thermally characterized by standard DSC, because a representative sample cannot be taken;
- $KClO_3$/Dodecane cannot be thermally characterized by standard DSC because of dodecane volatility;
- $KClO_4$/Dodecane cannot be thermally characterized by standard DSC because of dodecane volatility;
- H_2O_2/Flour cannot be thermally characterized by standard DSC because of H_2O_2 volatility;
- H_2O_2/Cumin cannot be thermally characterized by standard DSC because of H_2O_2 volatility;
- H_2O_2/Glycerin cannot be thermally characterized by standard DSC because of H_2O_2 volatility;
- H_2O_2/Flour cannot be characterized by ABL friction;
- H_2O_2/Cumin cannot be characterized by ABL friction;
- H_2O_2/Glycerin cannot be characterized by ABL friction;
- H_2O_2/Nitromethane cannot be characterized by ABL friction;
- AN is hard to characterize;
- AN/Gunpowder cannot be characterized by impact because of huge mismatch in particle size;
- AN/Gunpowder cannot be characterized by DSC because of huge mismatch in particle size affects sampling.

2.14 The future of the IDCA

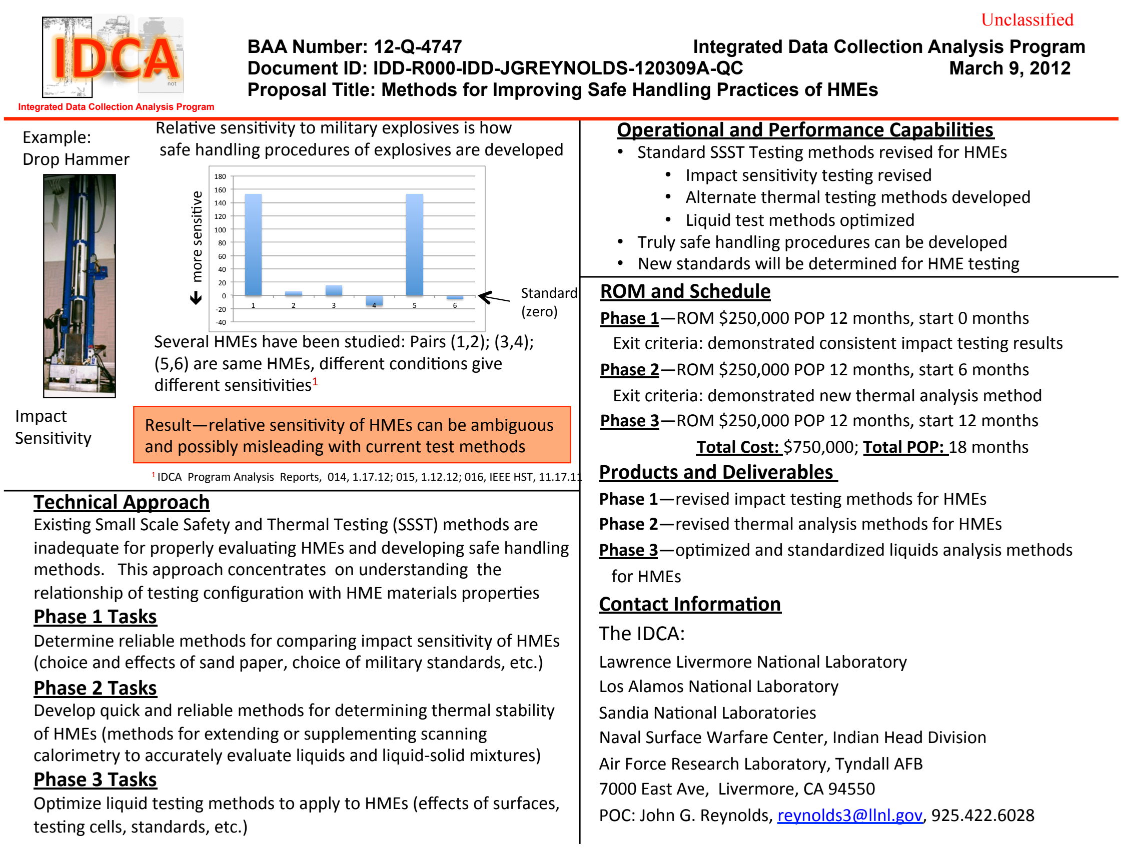


Figure 2.14.1. IDCA Quad chart for priority research areas in SSST testing of HMEs.

The results from the IDCA Proficiency Test illustrate the need for additional scientific research into SSST testing of HMEs. From the technical presentations and the conclusions, there are numerous topics that need resolution. Some priority topics are:

- Sandpaper in impact testing (composition—particle size);
- Standardization of positive/negative detection (instrumentation?);
- DSC of materials containing liquids and new thermal methods (volatility and reactivity);
- Revision of liquid testing methods (standards, with and without sandpaper, cavity drop);
- Standardization for HMEs that encompass all HME testing and handling issues (international group for standardization—IGUS; Explosives Testers User Group—ETUG);
- Resolution of statistical differences.

Figure 2.14.1 is an example of a proposal for additional research on recommended issues found by the IDCA from the Proficiency Test analyses. The research areas listed in the QUAD chart are only a few of the many issues encountered by the IDCA in the Proficiency Test that need further research to re-solve—how materials shift sensitivity in impact testing due to sandpaper grit size; revision of standard DSC testing methods to assure representative sampling of solid-solid and solid-liquid mixtures; opti-mizing liquid testing methods.

Figure 2.14.2. Call for papers for the APS-SCCM & AIRAPT-24 Joint Conference, Seattle, Washington USA July 7-12, 2013.

The IDCA is also organizing a session on testing issues in explosives safety evaluation. This session is to be at the APS-SCCM & AIRAPT-24 Joint Conference, Seattle, Washington USA July 7-12, 2013. This meeting is commonly referred to the Shock-Physics meeting that happens every two years. Figure 2.14.2 shows the call for papers.

In addition, there have been a great amount of scientific data, analyses and conclusions that need to be shared with the explosives community at large. Opportunities at this time that provide a mechanism for disseminating information are DTIC reports, professional meetings, and refereed publications. The IDCA members will continue to try to find additional sources of funding through proposal submission and public exposure.

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ABREVIATIONS, ACRONYMS AND INITIALISMS

-100	Solid separated through a 100-mesh sieve
ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQL
Al	Aluminum
AR	As received (separated through a 40-mesh sieve)

ARA	Applied Research Associates
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
C	Chemical symbol for carbon
CAS	Chemical Abstract Services registry number for chemicals
cm	centimeters
DH_{50}	The height the weight is dropped in Drop Hammer that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
DHS	Department of Homeland Security
DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analysis
ESD	Electrostatic Discharge
F_{50}	The weight or pressure used in friction test that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
fps	feet per second
H	Chemical symbol for hydrogen
H_2O	Chemical formulation for water
HME	homemade explosives or improvised explosives
HMX	Her Majesty's Explosive, cyclotetramethylene-tetranitramine
IDCA	Integrated Data Collection Analysis
IHD	Naval Surface Warfare Center, Indian Head Division
j	joules
$KClO_3$	Potassium Chlorate
$KClO_4$	Potassium Perchlorate
kg	kilograms
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
MBOM	Modified Bureau of Mines
N	Chemical symbol for nitrogen
$NaClO_3$	Sodium Chlorate
NSWC	Naval Surface Warfare Center
O	Chemical symbol for oxygen
PETN	Pentaerythritol tetranitrate
psig	pounds per square inch, gauge reading
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
RH	Relative humidity
RT	Room Temperature
RXQL	The Laboratory branch of the Airbase Sciences Division of the Materials & Manufacturing Directorate of AFRL
s	Standard Deviation
SEM	Scanning Electron Micrograph
Si	silicon
SNL	Sandia National Laboratories
SSST	small-scale safety and thermal
TGA	Thermogravimetric Analysis
TIL	Threshold level—level before positive event

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